

10458286

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PASSWORD:

TERMINAL (ENTER 1, 2, 3, OR ?):2

* * * * * Welcome to STN International * * * * *

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NEWS	8	DEC 15	MEDLINE update schedule for December 2004
NEWS	9	DEC 17	ELCOM reloaded; updating to resume; current-awareness alerts (SDIs) affected
NEWS	10	DEC 17	COMPUAB reloaded; updating to resume; current-awareness alerts (SDIs) affected
NEWS	11	DEC 17	SOLIDSTATE reloaded; updating to resume; current-awareness alerts (SDIs) affected
NEWS	12	DEC 17	CERAB reloaded; updating to resume; current-awareness alerts (SDIs) affected
NEWS	13	DEC 17	THREE NEW FIELDS ADDED TO IFIPAT/IFIUDB/IFICDB
NEWS	14	DEC 30	EPFULL: New patent full text database to be available on STN
NEWS	15	DEC 30	CAPLUS - PATENT COVERAGE EXPANDED
NEWS	16	JAN 03	No connect-hour charges in EPFULL during January and February 2005
NEWS	17	JAN 11	CA/CAPLUS - Expanded patent coverage to include Russia (Federal Institute of Industrial Property)
NEWS EXPRESS			JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005
NEWS HOURS			STN Operating Hours Plus Help Desk Availability
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NEWS LOGIN			Welcome Banner and News Items
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* * * * * STN Columbus * * * * *

10458286

FILE 'HOME' ENTERED AT 15:23:16 ON 25 JAN 2005

=> file registry
COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
0.21	0.21

FULL ESTIMATED COST

FILE 'REGISTRY' ENTERED AT 15:23:26 ON 25 JAN 2005
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Property values tagged with IC are from the ZIC/VINITI data file
provided by InfoChem.

STRUCTURE FILE UPDATES: 23 JAN 2005 HIGHEST RN 819046-01-0
DICTIONARY FILE UPDATES: 23 JAN 2005 HIGHEST RN 819046-01-0

TSCA INFORMATION NOW CURRENT THROUGH MAY 21, 2004

Please note that search-term pricing does apply when
conducting SmartSELECT searches.

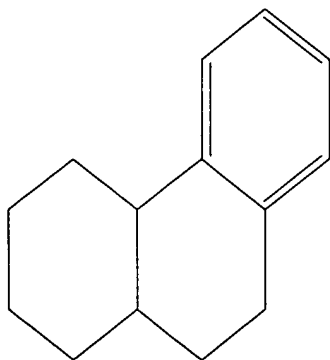
Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more
information enter HELP PROP at an arrow prompt in the file or refer
to the file summary sheet on the web at:
<http://www.cas.org/ONLINE/DBSS/registryss.html>

=>
Uploading C:\STNEXP4\QUERIES\10664165-ring.str

L1 STRUCTURE UPLOADED

=> d l1
L1 HAS NO ANSWERS
L1 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1 full
FULL SEARCH INITIATED 15:23:49 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - >1,000,000 TO ITERATE

< 23.0% PROCESSED 400000 ITERATIONS (1 INCOMPLETE) 33592 ANSWERS

10458286

INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.09

FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
BATCH **INCOMPLETE**

PROJECTED ITERATIONS: EXCEEDS 1000000
PROJECTED ANSWERS: EXCEEDS 144757

L2 33592 SEA SSS FUL L1

=> file caplus

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	161.33	161.54

FILE 'CAPLUS' ENTERED AT 15:24:07 ON 25 JAN 2005
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FILE COVERS 1907 - 25 Jan 2005 VOL 142 ISS 5
FILE LAST UPDATED: 24 Jan 2005 (20050124/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s l2

L3 5088 L2

=> d 5088 bib abs hitstr l3

L3 ANSWER 5088 OF 5088 CAPLUS COPYRIGHT 2005 ACS on STN

AN 1913:20380 CAPLUS

DN 7:20380

OREF 7:2947e-i,2948a

TI Perylene and its Derivatives. II

AU Weitzenbock, Richard; Seer, Christian

CS Univ. Graz.

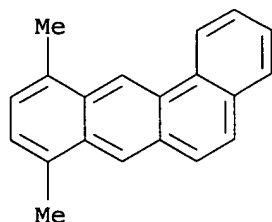
SO Ber. (1913), 46, 1994-2000

DT Journal

LA Unavailable

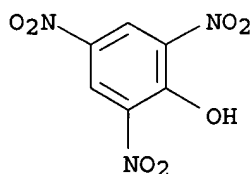
AB cf. C. A., 4, 2929.. (For nomenclature, see Bally and Scholl, C. A., 5, 3249). The yield of perylene can be increased to 4% by treating 1-C10H7Br with 3 parts of AlCl3 1 hr. at 140°. Under milder conditions (6 hrs. at 30-50°), from 20 g. C10H7Br were obtained traces of perylene; a small amount of a difficultly soluble substance, apparently a dinaphthyl-naphthalene, C30H20, yellowish leaflets, m. 282-3°; and about 1 g. of 2,2'-binaphthyl, possibly formed by rearrangement of 1,1'-binaphthyl under the influence of the AlCl3. β-Dinaphthylene oxide, heated 1 hr. at 140° with 4 parts of AlCl3, gives about 6%

10458286



CM 2

CRN 88-89-1
CMF C6 H3 N3 O7



L3 ANSWER 5011 OF 5088 CAPLUS COPYRIGHT 2005 ACS on STN
AN 1939:35193 CAPLUS
DN 33:35193
OREF 33:4981e-i,4982a-g
TI Syntheses in the phenanthrene series. II
AU Grewe, Rudolf
SO Ber. (1939), 72B, 785-90
DT Journal
LA Unavailable
GI For diagram(s), see printed CA Issue.
AB cf. C. A. 33, 3782.4. Continuation of the work on the synthesis on 9,13-disubstituted phenanthrene derivs. in connection with the study of the structure of morphine alkaloids. In the present paper are reported some new observations which confirm the structure assigned to the α,β -unsatd. dicarboxylic acid (I) described in part I; furthermore, there have been obtained phenanthrene-9-carboxylic acids with an ang-Me group at position 13. I heated with freshly distilled quinoline and Naturkupfer C 2 hrs. at 200° is decarboxylated chiefly to 2-(β -phenyl- α -carboxyethyl)methylenecyclohexane, PhCH₂CH(CO₂H)C₆H₉:CH₂ (II), m. 130°, and a smaller amount of an acid, m. 91° (probably formed from II by migration of the semicyclic double bond). Ozonolysis of II gave HCHO (isolated as the 2,4-dinitrophenylhydrazone (0.2 mol.), m. 162°), and the keto acid PhCH₂CH(CO₂H)CH.CO.CH₂.CH₂.CH₂.CH₂ (III), isolated as the semicarbazone (0.65 mol.) of the 174°-form of the Et ester. II undergoes ring closure with extraordinary ease, smoothly giving 2 compds. (IVa and IVb) which react with neither Br nor KMnO₄ and are ascribed the accompanying structure (in the original they are called 13-methyl-6,7,8,9,10,13,14-hexahydrophenanthrene-9-carboxylic acids but they are evidently the 5,6,7,8,9,10,13,14-octahydro compds.). On heating with Pd sponge up to 280°, which ordinarily dehydrogenates similar phenanthrene derivs. without an ang-Me group in a few min., they are hardly attacked, and only after long heating at about 320° could a small amount of an aromatic hydrocarbon, definitely identified as phenanthrene, be isolated. The

intermediate carboxylic acid is naturally at once decarboxylated at the temperature of the reaction. The recovered non-dehydrogenated material is always a mixture of IVa and IVb even when either of the pure isomers is used as starting material. None other of the total 4 possible racemates has thus far been obtained. Several attempts to effect complete aromatization of IVa and IVb with Se were unsuccessful. A vigorous reaction occurs but the crystalline products are not phenanthrenes; they form no addition product

with

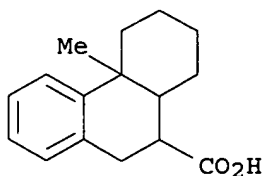
picric acid. A simplification of the synthesis of the IV by means of the Grignard reaction suggested itself. The ester of III can be converted with MeMgI into the lactone (V) only under definite conditions. During the reaction the primarily formed tertiary HO ester splits off EtOH. The yield is satisfactory and can be increased by starting directly from the free acid (III) and using 2 mols. MeMgI. By carefully working up the product it is possible to isolate the crystalline HO acid, PhCH₂CH(CO₂H)C₆H₉(OH)Me (VI), which, however, changes into V with extraordinary ease on gentle warming in ether. The lactone ring in V is only slowly opened by alkalis; it has thus far not been possible to prepare an ester of VI. The opening of the lactone ring is not necessary for the subsequent cyclization; with H₃PO₄ at 50°, V gives IVa and IVb with extraordinary ease. The striking ease of this rearrangement of V, which is so resistant toward other forms of attack, cannot be explained by the assumption alone of intermediate unsatd. acids. As V is easily soluble in cold H₃PO₄, probably phosphoric esters of HO acids of type VI have a decisive influence on the ring closure. V heated with P₂O₅ gives an entirely different result. There is formed a hydrocarbon C₁₅H₁₈ (VII), smoothly dehydrogenated to 1-methylphenanthrene, m. 119° (picrate, m. 135-6°) by heating with Pd. Since the Me group cannot have migrated under the mild conditions of the dehydrogenation, VII must have structure VIII or that of the Δ^{11,12}-isomer. The ultraviolet absorption spectrum, with maximum at 254 and 265 mμ, makes it probable that VII is a mixture of the 2 isomers. IVa and IVb, from II and sirupy H₃PO₄ at 80°, m. 142° and 178°, resp.

2-(β-Phenyl-α-carboxyethyl)-1-methylcyclohexanol (VI) (15 g. from 18 g. III in ether at 0° treated slowly with the Grignard reagent from 26 g. MeI, allowed to stand several hrs. at room temperature, heated some hrs. on the water bath and decomposed with much ice and dilute HCl), m. 121° (foaming). V (16 g. from 21 g. of the Et ester of III treated at 0° with the Grignard reagent from 8.5 cc. MeI in the course of several hrs., allowed to stand overnight at 0°, decomposed with ice and dilute HCl and, after evaporation of the ether, boiled 30 min. in 250 cc. alc. with 25 g. Girard reagent T and 25 cc. AcOH), b_{0.2} 167°. 1-Methylhexahydrophenanthrene (VII), b_{0.3} 124°.

IT 798557-11-6, 9-Phenanthrenecarboxylic acid, 4b,5,6,7,8,8a,9,10-octahydro-4b-methyl-
(isomers)

RN 798557-11-6 CAPLUS

CN 9-Phenanthrenecarboxylic acid, 4b,5,6,7,8,8a,9,10-octahydro-4b-methyl-
(4CI) (CA INDEX NAME)



10458286

Connecting via Winsock to STN

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NEWS	16	JAN 03	No connect-hour charges in EPFULL during January and February 2005
NEWS	17	JAN 11	CA/CAPLUS - Expanded patent coverage to include Russia (Federal Institute of Industrial Property)
NEWS EXPRESS			JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005
NEWS HOURS			STN Operating Hours Plus Help Desk Availability
NEWS INTER			General Internet Information
NEWS LOGIN			Welcome Banner and News Items
NEWS PHONE			Direct Dial and Telecommunication Network Access to STN
NEWS WWW			CAS World Wide Web Site (general information)

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* * * * * STN Columbus * * * * *

10458286

FILE 'HOME' ENTERED AT 15:23:16 ON 25 JAN 2005

=> file registry
COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
0.21	0.21

FULL ESTIMATED COST

FILE 'REGISTRY' ENTERED AT 15:23:26 ON 25 JAN 2005
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STRUCTURE FILE UPDATES: 23 JAN 2005 HIGHEST RN 819046-01-0
DICTIONARY FILE UPDATES: 23 JAN 2005 HIGHEST RN 819046-01-0

TSCA INFORMATION NOW CURRENT THROUGH MAY 21, 2004

Please note that search-term pricing does apply when
conducting SmartSELECT searches.

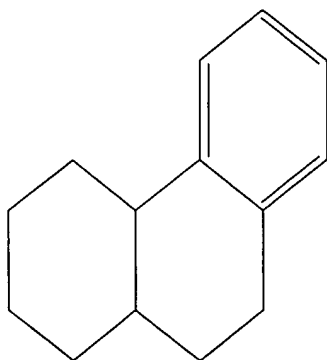
Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more
information enter HELP PROP at an arrow prompt in the file or refer
to the file summary sheet on the web at:
<http://www.cas.org/ONLINE/DBSS/registryss.html>

=>
Uploading C:\STNEXP4\QUERIES\10664165-ring.str

L1 STRUCTURE UPLOADED

=> d l1
L1 HAS NO ANSWERS
L1 STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1 full
FULL SEARCH INITIATED 15:23:49 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - >1,000,000 TO ITERATE

< 23.0% PROCESSED 400000 ITERATIONS (1 INCOMPLETE) 33592 ANSWERS

10458286

INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.09

FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
BATCH **INCOMPLETE**

PROJECTED ITERATIONS: EXCEEDS 1000000
PROJECTED ANSWERS: EXCEEDS 144757

L2 33592 SEA SSS FUL L1

=> file caplus

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	161.33	161.54

FILE 'CAPLUS' ENTERED AT 15:24:07 ON 25 JAN 2005
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
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FILE COVERS 1907 - 25 Jan 2005 VOL 142 ISS 5
FILE LAST UPDATED: 24 Jan 2005 (20050124/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s l2

L3 5088 L2

=> d 5088 bib abs hitstr l3

L3 ANSWER 5088 OF 5088 CAPLUS COPYRIGHT 2005 ACS on STN

AN 1913:20380 CAPLUS

DN 7:20380

OREF 7:2947e-i,2948a

TI Perylene and its Derivatives. II

AU Weitzenbock, Richard; Seer, Christian

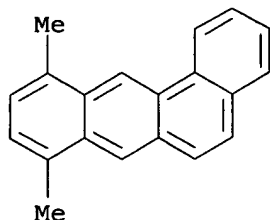
CS Univ. Graz.

SO Ber. (1913), 46, 1994-2000

DT Journal

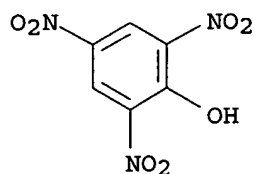
LA Unavailable

AB cf. C. A., 4, 2929.. (For nomenclature, see Bally and Scholl, C. A., 5, 3249). The yield of perylene can be increased to 4% by treating 1-C10H7Br with 3 parts of AlCl3 1 hr. at 140°. Under milder conditions (6 hrs. at 30-50°), from 20 g. C10H7Br were obtained traces of perylene; a small amount of a difficultly soluble substance, apparently a dinaphthyl naphthalene, C30H20, yellowish leaflets, m. 282-3°; and about 1 g. of 2,2'-binaphthyl, possibly formed by rearrangement of 1,1'-binaphthyl under the influence of the AlCl3. β -Dinaphthylene oxide, heated 1 hr. at 140° with 4 parts of AlCl3, gives about 6%



CM 2

CRN 88-89-1
CMF C6 H3 N3 O7



L3 ANSWER 5011 OF 5088 CAPLUS COPYRIGHT 2005 ACS on STN
 AN 1939:35193 CAPLUS
 DN 33:35193
 OREF 33:4981e-i,4982a-g
 TI Syntheses in the phenanthrene series. II
 AU Grewe, Rudolf
 SO Ber. (1939), 72B, 785-90
 DT Journal
 LA Unavailable
 GI For diagram(s), see printed CA Issue.
 AB cf. C. A. 33, 3782.4. Continuation of the work on the synthesis on 9,13-disubstituted phenanthrene derivs. in connection with the study of the structure of morphine alkaloids. In the present paper are reported some new observations which confirm the structure assigned to the α,β -unsatd. dicarboxylic acid (I) described in part I; furthermore, there have been obtained phenanthrene-9-carboxylic acids with an ang-Me group at position 13. I heated with freshly distilled quinoline and Naturkupper C 2 hrs. at 200° is decarboxylated chiefly to 2-(β -phenyl- α -carboxyethyl)methylenecyclohexane, PhCH₂CH(CO₂H)C₆H₉:CH₂ (II), m. 130°, and a smaller amount of an acid, m. 91° (probably formed from II by migration of the semicyclic double bond). Ozonolysis of II gave HCHO (isolated as the 2,4-dinitrophenylhydrazone (0.2 mol.), m. 162°), and the keto acid PhCH₂CH(CO₂H)CH.CO.CH₂.CH₂.CH₂.CH₂ (III), isolated as the semicarbazone (0.65 mol.) of the 174°-form of the Et ester. II undergoes ring closure with extraordinary ease, smoothly giving 2 compds. (IVa and IVb) which react with neither Br nor KMnO₄ and are ascribed the accompanying structure (in the original they are called 13-methyl-6,7,8,9,10,13,14-hexahydrophenanthrene-9-carboxylic acids but they are evidently the 5,6,7,8,9,10,13,14-octahydro compds.). On heating with Pd sponge up to 280°, which ordinarily dehydrogenates similar phenanthrene derivs. without an ang-Me group in a few min., they are hardly attacked, and only after long heating at about 320° could a small amount of an aromatic hydrocarbon, definitely identified as phenanthrene, be isolated. The

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with

picric acid. A simplification of the synthesis of the IV by means of the Grignard reaction suggested itself. The ester of III can be converted with MeMgI into the lactone (V) only under definite conditions. During the reaction the primarily formed tertiary HO ester splits off EtOH. The yield is satisfactory and can be increased by starting directly from the free acid (III) and using 2 mols. MeMgI. By carefully working up the product it is possible to isolate the crystalline HO acid, PhCH₂CH(CO₂H)C₆H₉(OH)Me (VI), which, however, changes into V with extraordinary ease on gentle warming in ether. The lactone ring in V is only slowly opened by alkalies; it has thus far not been possible to prepare an ester of VI. The opening of the lactone ring is not necessary for the subsequent cyclization; with H₃PO₄ at 50°, V gives IVa and IVb with extraordinary ease. The striking ease of this rearrangement of V, which is so resistant toward other forms of attack, cannot be explained by the assumption alone of intermediate unsatd. acids. As V is easily soluble in cold H₃PO₄, probably phosphoric esters of HO acids of type VI have a decisive influence on the ring closure. V heated with P₂O₅ gives an entirely different result. There is formed a hydrocarbon C₁₅H₁₈ (VII), smoothly dehydrogenated to 1-methylphenanthrene, m. 119° (picrate, m. 135-6°) by heating with Pd. Since the Me group cannot have migrated under the mild conditions of the dehydrogenation, VII must have structure VIII or that of the Δ^{11,12}-isomer. The ultraviolet absorption spectrum, with maximum at 254 and 265 mμ, makes it probable that VII is a mixture of the 2 isomers. IVa and IVb, from II and sirupy H₃PO₄ at 80°, m. 142° and 178°, resp.

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IT 798557-11-6, 9-Phenanthrenecarboxylic acid, 4b,5,6,7,8,8a,9,10-octahydro-4b-methyl-
(isomers)

RN 798557-11-6 CAPLUS

CN 9-Phenanthrenecarboxylic acid, 4b,5,6,7,8,8a,9,10-octahydro-4b-methyl-
(4CI) (CA INDEX NAME)

